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DECLARATION UNDER 37 C.F.R. § 1.132

I, Dr. Won nam Kang, do declare and state that:

I am a co-inventor of the above-identified application.

I received a Bachelor of Science in Physics in August 1987, a Master of Science in Physics in February 1990, and a Ph.D. in Physics in February 1994 from the Sungkyunkwan University in South Korea.

I am presently a Research Associate Professor at the Pohang University of Science and Technology in South Korea. I worked for the University of Kansas as a Post-Doctor from September 1995 through July 1996, and the University of Houston as a Research Associate Professor from July 1996 through May 1998.

A. THE APPLIED REFERENCE

I have examined the patent application of Won nam Kang et al., Application No. 10/097,975, filed March 15, 2002 including the Amendment filed September 10, 2003 under 37 C.F.R. § 1.111. I have also examined the final Office Action of the Patent

Office dated November 20, 2003 and, in particular, the Finnemore et al. reference (U.S. Patent No. 6,514,557).

As a result of my detailed review of the above items, I would like to make the following observations.

U.S. Patent No. 6,514,557 (Finnemore)

Finnemore is directed to a method of manufacturing a *magnesium diboride wire*. The magnesium diboride wire is obtained by reacting a boron object with magnesium vapor at or near 950°C for approximately two hours. See, e.g., Fig. 1a.

B. THE PRESENT INVENTION

In the present invention, the sole pending claim 1 provides a method of manufacturing a *magnesium diboride thin film*. Claim 1 further provides that a substrate with boron thin film and a magnesium source are placed inside a heat source, rapidly heated for 10-60 minutes, and then cooled. The heating temperature is equal to or greater than 600°C and less than 950°C. This rapid thermal process can effectively prevent degradation of the magnesium diboride thin film caused by chemical reaction with the substrate underlying the magnesium diboride thin film.

C. DISCUSSION

After carefully reviewing the Finnemore reference cited by the Patent Office, in my opinion, the present invention, as set forth in claim 1, is not anticipated by or obvious in view of the Finnemore reference. A detailed explanation for my conclusion follows.

I conducted an experimentation in the same manner described in the article entitled, "Growth of superconducting MgB_2 thin films via postannealing techniques," W. N. Kang et al., Physica C, pp. 24-30 (2003), per copy enclosed.

Figure 4 of the article illustrates magnetic field dependence of critical current density (J_c) at 5 K for samples M700C30m, M800C30m, M900C30m, M950C30m, M900C60m, and M900C120m. J_c is a very important factor for use in practical applications of high-field superconducting magnet systems.

M900C120m and M950C30m are samples of the Finnemore reference, and M700C30m, M800C30m, M900C30m and M900C60m are samples of the present invention. Referring again to Figure 4, the J_c values of samples M900C120m and M950C30m are approximately 6 MA/cm^2 at zero field, and the J_c value of sample M900C30m is approximately 20A/cm^2 at zero field. As such, the J_c values of the samples of the present invention at zero field are approximately three times greater than those of the Finnemore reference. Accordingly, as the value of J_c at zero field increases, the superconducting characteristics of the samples are improved.

The magnetic field dependence of J_c of the samples shows different behavior. The samples annealed at less than 950°C for a short period of time (e.g., 30 minutes), show weak-field dependence compared to samples M900C120m and M950C30m. Sample M950C30m shows a very dense surface morphology highly oriented along the c-axis when compared to sample M900C120m. The strong-field dependance of J_c implies that the sample contains fewer pinning sites than M900C30m. The J_c data supports the fabrication process for sample M900C30m according to the present invention being the optimal condition for large-scale applications.

Also, referring to Figure 3 of the article, the superconducting transition temperature (T_c) and transition width (ΔT_c) depends greatly on the annealing temperature and time. In general, the superconducting characteristic improves as T_c increases and ΔT_c decreases. Specifically, in Figure 3(b), the sample annealed for 120

minutes shows a lower T_c and a broad superconducting transition indicating that longer annealing degrades superconductivity by changing the growth orientation of MgB_2.

In view of the foregoing reasons, I believe that the present invention as set forth in claim 1 is not anticipated by or obvious in view of the Finnemore reference.

D. CONCLUSION

I declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Date: Mar. 11, 2004

WNKang
Dr. Won nam Kang

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Growth of superconducting MgB_2 thin films via postannealing techniques

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Abstract

We report the effect of annealing on the superconductivity of MgB_2 thin films as a function of the postannealing temperature in the range from 700 to 950 °C and of the postannealing time in the range from 30 to 120 min. On annealing at 900 °C for 30 min, we obtained the best-quality MgB_2 films with a transition temperature of 39 K and a critical current density of $\sim 10^7$ A/cm^2. Using scanning electron microscopy, we also investigated the film growth mechanism. The samples annealed at higher temperatures showed larger grain sizes, well-aligned crystal structures with preferential orientation along the *c*-axis, and smooth surface morphologies. However, a longer annealing time prevented the alignment of grains and reduced superconductivity, indicating a strong interfacial reaction between the substrate and the MgB_2 film.

PACS: 74.72.Yg; 74.76.–w
Keywords: Thin film growth; Annealing; Critical current; Grain alignment

1. Introduction

The discovery of superconductivity in the MgB_2 compound [1], with the highest transition temperature (T_c) of 39 K among the metallic superconductors, has generated great interest in both basic science [2–9] and practical applications [10–17]. Recently, the two-gap nature of MgB_2 superconductor has been confirmed by using scanning tunneling microscopy on highly *c*-axis oriented thin films. The strongly linked nature of the intergrains [11] with metallic transport properties [4–9] and the relatively simple crystal structure of this material are further advantages of its use in technological applications. An upper critical field (H_{c2}) of 29–39 T [4,10], which is much higher than those of conventional superconductors, was observed, suggesting that MgB_2 would be a very promising compound for large-scale application in superconducting solenoids using mechanical cryocoolers, such as closed cycle refrigerators. In addition to its higher T_c and H_{c2}, the magnitude of the critical current density (J_c) is also a very important factor for use in practical applications. In the case of MgB_2 thin films with preferred orientations along the *c*-axis, a very high-current carrying capability, $J_c \sim 10^7$ A/cm^2 at 5 K, was observed [9], which is two orders of magnitude larger than the

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values for the Fe-clad MgB_2 wires prepared by using powder in tube techniques [12–15].

Since the successful growth of MgB_2 thin films [14], a number of groups have intensively conducted experiments to fabricate high-quality films [17–28]. Two-step (ex situ) techniques, which require Mg diffusion into pure B films at high temperatures, have been quite successful in growing high-quality films [15–18]. However, the surface quality for electronic-device applications is still far from being achieved. Nevertheless, very remarkable research results concerning electronic-device applications, such as superconducting quantum interference devices (SQUID) and microwave devices, have been reported [28–31]. Brinkman et al. [28] reported successful fabrication of MgB_2 SQUID. Using low-T_c thin films ($T_c \sim 22$ K), they obtained a nice voltage modulation up to 19 K, showing that high-temperature SQUID operating above 30 K could be developed by using metallic superconductors if higher-T_c MgB_2 films are used. Moreover, Jin et al. [30] observed a very low surface resistance of 19 $\mu\Omega$cm at 7.2 GHz and 4.2 K in MgB_2 films, which is comparable with that of a high-quality $YBa_2Cu_3O_7$ thin film. This is a very surprising result since MgB_2 films have a rough surface morphology compared to $YBa_2Cu_3O_7$ thin films. Very recently, in situ growth of high-quality MgB_2 thin films by using a hybrid physical–chemical vapor deposition technique, has been reported by Zeng et al. [32]. This is believed to be a very promising growth technique in terms of widespread applications for superconducting electronics, such as multilayer Josephson junctions and digital circuits.

In this paper, we report the superconducting properties and the growth mechanism of MgB_2 thin films prepared under various annealing temperatures and times. The films annealed at 900 °C for 30 min showed the best superconductivity whereas the samples annealed at 950 °C for 30 min revealed smooth and compact surface morphologies with preferential orientation along the c-axis. The present study was possible since we used a pulsed laser deposition (PLD) system attached to a load-lock sample preparation chamber, which routinely produced high-quality MgB_2 thin films with reliable reproducibility.

2. Experimental

The MgB_2 thin films were grown on r-cut Al_2O_3 $(1\,\bar{1}\,0\,2)$ single crystals under high-vacuum conditions of $\sim 10^{-7}$ Torr by using PLD and postannealing techniques, reported in our earlier paper [9,16]. The PLD system used in the present study is especially designed for the growth of metallic thin films, which should not be contaminated by O_2 or H_2O. Furthermore, this system is able to transfer the precursor B films from the growth chamber to a clean dry box without exposure to air. We found that the preparation of pure B film was very crucial for fabrication of high-quality c-axis-oriented MgB_2 thin films. The laser energy density used in this study was 10–20 J/cm^2 at a laser flux of 450 mJ/pulse and a pulse frequency of 8 Hz. After precursor B films had been deposited, it was placed in a Nb tube together with high-purity Mg metal (99.9%), and the tube was sealed by using an arc welder in an Ar atmosphere. Postannealing was carried out at several temperatures from 700 to 950 °C and at several annealing times from 30 to 120 min. The film growth conditions and transport properties are summarized in Table 1. Typical dimensions of the samples were 10 mm in length, 10 mm in width and 0.4–0.5 μm in thickness.

The resistivity was measured by the standard dc four-probe method after cutting the samples into rectangular shapes of size 1 mm × 5 mm. To obtain good ohmic contacts, we deposited Au contact pads after cleaning the film surface with Ar-ion beam milling. This process does not degrade the superconducting properties of MgB_2 thin films. The magnetization was measured by using SQUID magnetometry. The surface morphology was studied by using field-emission scanning electron microscopy (SEM).

3. Results and discussion

Fig. 1(a)–(f) shows SEM pictures for various MgB_2 films annealed for 30 min at (a) 800 °C, (b) 850 °C, (c) 900 °C, and (d) 950 °C, and annealed at 900 °C for (e) 60 min and (f) 120 min. Sub-micron sized MgB_2 single crystals with hexagonal shape can be observed. We can see clearly

Table 1
Summary of the film growth conditions, annealing temperature (T_{ann}) and annealing time (t_{ann}), and the transport properties of superconducting transition temperature (T_c), transition width (ΔT_c), normal-state resistivity ($\rho_{290\ K}$) at 290 K, and residual resistivity ratio (RRR = $\rho_{40\ K}/\rho_{290\ K}$)

Sample ID	T_{ann} (K)	t_{ann} (min)	T_c (K)	ΔT_c (K)	$\rho_{290\ K}$ (μΩ cm)	RRR
M700C30m	700	30	38.1	2.3	68	1.4
M800C30m	800	30	37.5	0.4	75	1.5
M850C30m	850	30	37.4	0.7	49	1.7
M900C30m	900	30	39.0	0.3	14	2.3
M950C30m	950	30	37.5	0.3	24	2.1
M900C60m	900	60	39.1	0.6	18	2.9
M900C120m	900	120	38.3	1.5	20	2.1

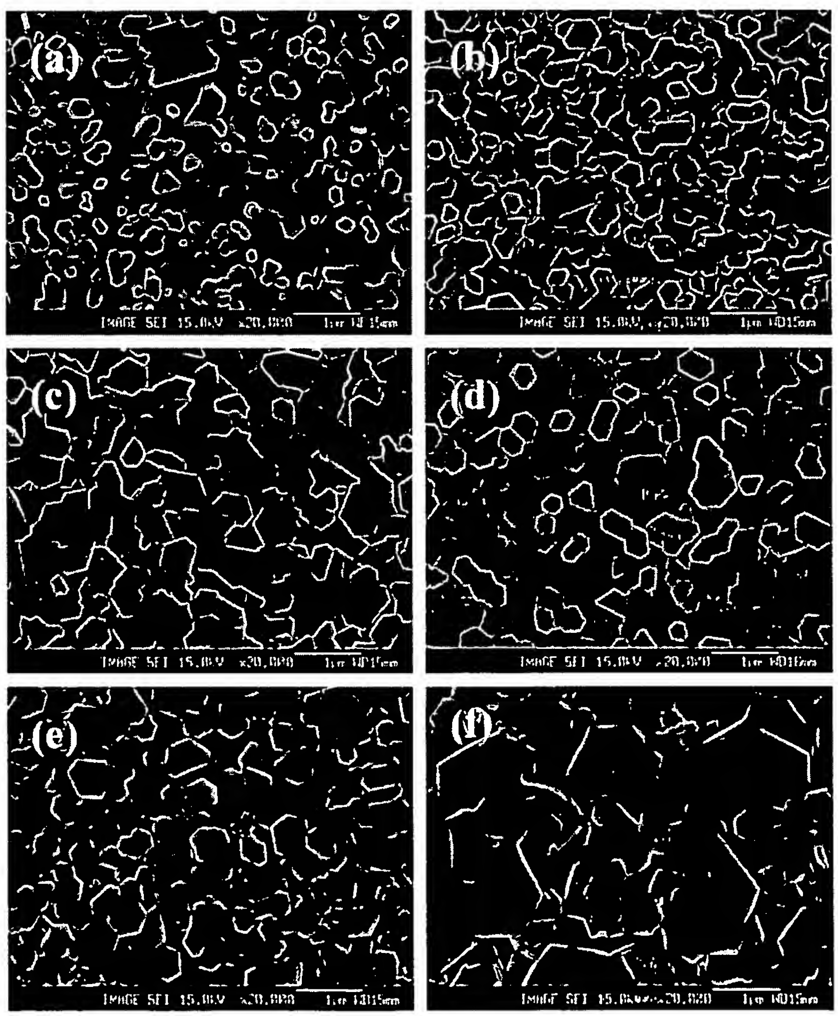

Fig. 1. SEM pictures of MgB_2 films annealed for 30 min at (a) 800 °C, (b) 850 °C, (c) 900 °C, and (d) 950 °C and annealed at 900 °C for (e) 60 min and (f) 120 min.

that the grain connectivity becomes very strong as the annealing temperature is increased. The higher temperature also assists to form preferred orientation along the *c*-axis, which is perpendicular to the substrate surface, whereas annealing times longer than 60 min at 900 °C hinders the alignment of grains so that a polycrystalline surface morphology is revealed, as shown in Fig. 1(e) and (f). It is very interesting that MgB_2 thin films grow with preferred orientation on Al_2O_3 even though the lattice-matching relationship between MgB_2 and the substrate is not well satisfied. Recently, Tian et al. [25] reported an interesting result based on interfacial reaction between an MgB_2 film and the Al_2O_3 substrate. They found intermediate epitaxial layers of MgO and $MgAl_2O_4$ in the film/substrate interface, demonstrating that epitaxial growth of MgB_2 on an Al_2O_3 substrate is possible. Our results further suggest that MgB_2 thin films grow along a favorable direction in a wide temperature window of 850–950 °C and for annealing times around 30 min. If the polycrystalline growth at longer annealing times shown in Fig. 1(e) and (f) is to be understood, further microscopic experiments addressing the crystallographic relations of MgB_2 on Al_2O_3 substrates is required.

The temperature dependence of the resistivity of the MgB_2 films after annealing (a) at 700–950 °C for 30 min and (b) at 900 °C for 30–120 min, is shown in Fig. 2, where the resistivity is normalized to its value at 290 K. The transport properties extracted from these data are listed in Table 1. All samples show higher values for T_c, 37.4–39.1 K, whereas the transition width (ΔT_c) varies significantly with annealing conditions. Samples annealed at 900 °C for 30 min show smaller ΔT_c and $\rho_{290\ K}$; thus, this annealing condition can be considered as an optimum growth process. As the annealing temperature is increased up to 900 °C, we can see a gradual increase in RRR and a dramatic decrease in the normal-state resistivity, indicating that grain connectivity becomes very strong with increasing temperature, as indicated in the SEM images in Fig. 1. However, M950C30m has a much higher resistivity than M900C30m although the SEM image shows strong connectivity of the grain boundary. This suggests that an interfacial reaction between the substrate and the

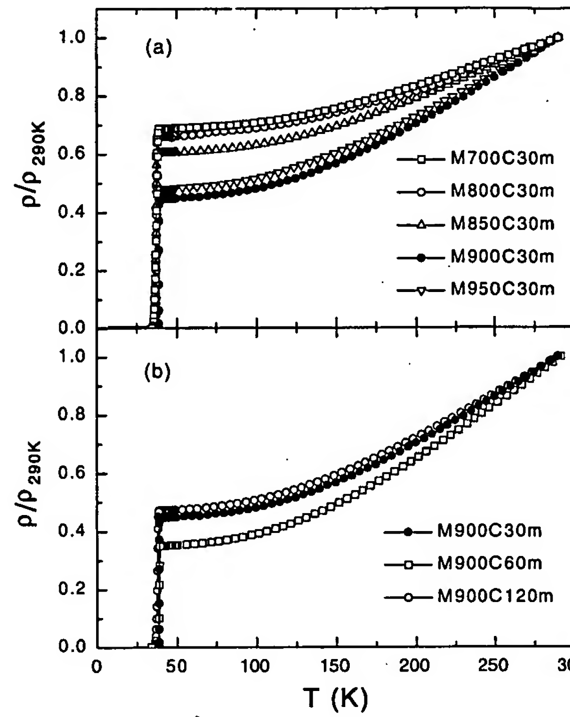

Fig. 2. Temperature dependence of the resistivity for MgB_2 films after annealing (a) at 700–950 °C for 30 min and (b) at 900 °C for 30–120 min.

MgB_2 film can result from high-temperature annealing at temperatures above 900 °C. For the sample annealed at 900 °C, the resistivity increases gradually with increasing annealing time from 30 to 120 min, which is consistent with the polycrystalline surface morphology shown in Fig. 1(e) and (f).

Fig. 3 shows the temperature dependence of the zero-field-cooled magnetization data at 10 Oe for the samples fabricated (a) at 700–950 °C for 30 min and (b) at 900 °C for 30–120 min. Regardless of the growth condition, all samples show bulk diamagnetism at temperatures below 10 K. For the samples annealed for 30 min, we can see a systematic enhancement of T_c with increasing annealing temperature up to 900 °C. The T_c of M950C30m is lower than that of M900C30m, but is comparable to that of single crystalline MgB_2. Considering the sharp ΔT_c of ~0.3 K for M950C30m and its well-grown surface morphology, as shown in Fig. 1(d), M950C30m may be a

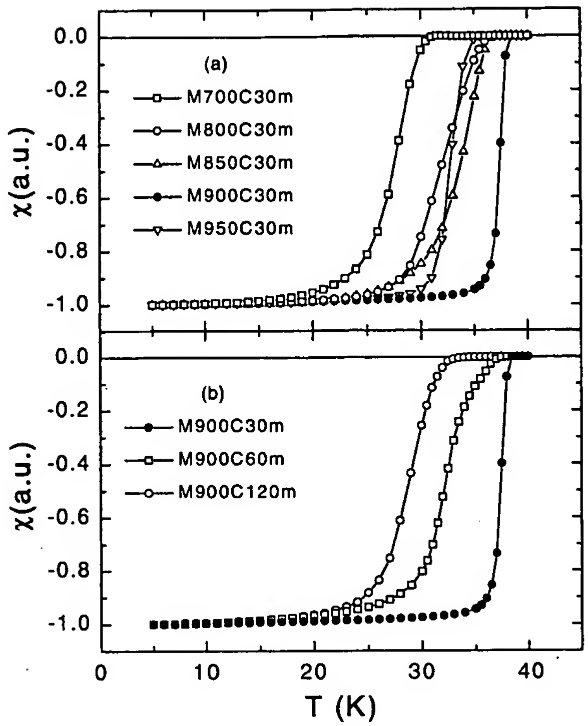

Fig. 3. Temperature dependence of zero-field-cooled magnetization data at 10 Oe for the films fabricated at (a) 700–950 °C for 30 min and (b) 900 °C for 30–120 min.

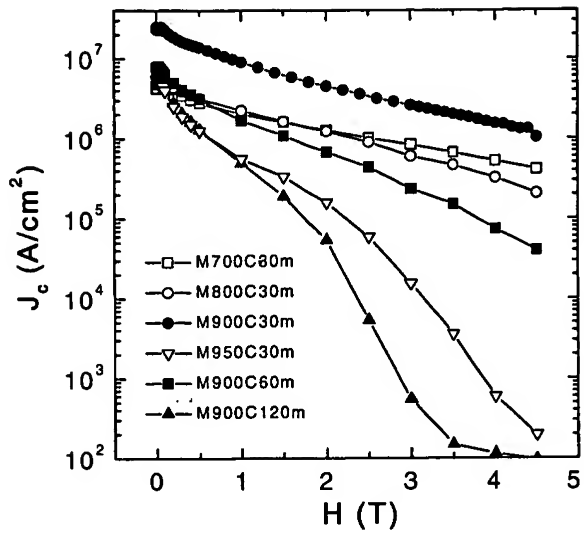

Fig. 4. Magnetic field dependence of J_c at 5 K for various films grown at 700–950 °C for 30 min and at 900 °C for 30–120 min.

good sample for studying physical properties. However, M900C30m better satisfies the requirements for practical applications because of its higher T_c and J_c (see Fig. 4). Using M950C30m, indeed, decisive experimental result was obtained by Iavarone et al. [2], confirming the two-gap nature of the MgB_2 superconductor. In Fig. 3(b), the samples annealed for 60–120 min show a lower T_c and a broad superconducting transition, indicating that longer annealing degrades superconductivity by changing the growth orientation of MgB_2 grains, as shown in the SEM images.

Fig. 4 shows the magnetic field dependence of J_c at 5 K for M700C30m, M800C30m, M900C30m, M950C30m, M900C60m, and M900C120m. To estimate the critical current density, we measured the magnetic field dependence of magnetization (M–H) loop. The sample size, $5 \times 3 - 4$ mm^2, rather than the grain size, was used to evaluate J_c by using Bean's model [33]. M900C30m had the highest J_c of all the samples, 2.5×10^7 A/cm^2 at 5 K in zero field, which is comparable to the values in previous reports on MgB_2 films [7,19,32] and on high-T_c cuprates [34,35]. A J_c of $\sim 10^6$ A/cm^2 at 4.5 T is sufficiently high for practical applications of high-field superconducting magnet systems. This result reflects that the grains of M900C30m are connected very strongly with a high density of pinning sites, such as stacking faults, dislocations, and grain boundaries. At zero field, the J_c values for the other films are $\sim 6 \times 10^6$ A/cm^2 at zero field, but the magnetic field dependence of J_c shows quite different behavior. The samples annealed at equal or below 900 °C and for shorter time (30 min), show weak-field dependence compared to the samples of M950C30m and M900C120m. As shown in the SEM image (Fig. 1(f)), M900C120m has a polycrystalline structure; thus, the J_c depends strongly on the magnetic field as with polycrystalline Fe-clad MgB_2 wires [13]. Different from M900C120m, however, M950C30m shows a very dense surface morphology highly oriented along the c-axis as shown in Fig. 1(d). In the case of this film, the strong-field dependence of J_c probably implies that this sample contains fewer pinning sites than M900C30m (Fig. 1(c)). Our J_c data further support the fabrication process for M900C30m sample being the optimal condition for large-scale applications. For electronic-device

application, M950C30m is favored, but we should reduce the interfacial reaction at high temperatures. A solution for this difficulty is to use an in situ low-temperature process [26,27,32], relying on chemically stable substrates or buffer layers.

4. Summary

The superconducting properties and the growth mechanism of MgB_2 thin films prepared under various annealing temperatures and times were investigated. The films annealed at 900 °C for 30 min showed the best superconductivity with a transition temperature of 39 K and a critical current density of $\sim 10^7$ A/cm^2 at 5 K and zero field. On the other hand, the samples annealed at 950 °C for 30 min revealed smooth and compact surface morphologies with preferential orientations along the *c*-axis. We found that MgB_2 thin films grow along a preferred direction in the annealing temperature window of 850–950 °C and for annealing times around 30 min. By using a PLD system attached to a load-lock sample preparation chamber, we are able to fabricate very high-quality MgB_2 thin films with reliable reproducibility. Our results suggest that an in situ low-temperature process should be developed in order to apply MgB_2 thin films to superconducting electronics.

Acknowledgements

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